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IS 9406 (1980): Calcium Silicate for Rubber [PCD 13: Rubber and Rubber Products]



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IS : 9406 - 1980

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“पुनर्पठ १९९५”

“RE-AFFIRMED 1995”

SPECIFICATION FOR  
CALCIUM SILICATE FOR RUBBER INDUSTRY

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# Indian Standard

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# *Indian Standard*

## SPECIFICATION FOR CALCIUM SILICATE FOR RUBBER INDUSTRY

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 5 January 1980, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

**0.2** Calcium silicate is an important filler. It is, however, essential that it should be of proper quality so that through it no unwarranted impurities are added to the vulcanizate.

**0.3** This standard contains clauses **2.3** and **3.1** which call for agreement between the purchaser and the supplier.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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### 1. SCOPE

**1.1** This standard prescribes the requirements and methods of sampling and test for calcium silicate for rubber industry.

### 2. REQUIREMENTS

**2.1 Description** — The material shall be white amorphous powder. It shall be dry and free from extraneous impurities and grit.

**2.2** The material shall also comply with the requirements laid down in Table 1, when tested according to the method indicated against each.

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\*Rules for rounding off numerical values (revised).

**TABLE 1 REQUIREMENTS FOR CALCIUM SILICATE FOR RUBBER INDUSTRY**

( Clause 2.2 )

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix	Cl No. in IS : 7086 ( Part I )- 1973*
(1)	(2)	(3)	(4)	(5)
i)	Bulk density, g/ml	$0.16 \pm 0.03$	A	—
ii)	Sieve residue, percent by mass, <i>Max</i>		—	3
	a) Through 53-micron Sieve	5		
	b) Through 75-micron Sieve	2		
	c) Through 150-micron Sieve	1		
iii)	Relative density 27/27°C	$1.97 \pm 0.05$	—	4
iv)	pH	8 to 9	—	5
v)	Moisture content, percent by mass	$3.5 \pm 0.5$	—	7
vi)	Matter soluble in water, percent by mass, <i>Max</i>	0.5	—	8
vii)	Loss on ignition, percent by mass	$12 \pm 1.5$	—	10
viii)	Manganese ( as Mn ), percent by mass, <i>Max</i>	0.001 2	—	11
ix)	Copper ( as Cu ), percent by mass, <i>Max</i>	0.001 8	—	12
x)	Lead ( as Pb ), percent by mass, <i>Max</i>	0.001	—	14
xi)	Calcium oxide, percent by mass, <i>Max</i>	$18 \pm 2$	B	—
xii)	Silica ( as SiO <sub>2</sub> ), percent by mass	$68 \pm 4$	C	—
xiii)	Oil absorption, g of linseed oil per 100 g of the material	$130 \pm 10$	D	—
xiv)	Sedimentation volume in toluene, ml	$15 \pm 1$	E	—
xv)	Retention in suspension, percent by mass, <i>Min</i> :		F	—
	a) After 5 minutes	80		
	b) After 20 minutes	60		

\*Methods of sampling and test for rubber compounding ingredients, Part I.



**2.3 Compounding Test** — If desired by the purchaser, the material may be compounded in styrene butadiene rubber test recipe and the properties of calcium silicate compared with the approved sample.

### 3. PACKING AND MARKING

**3.1 Packing** — The material shall be packed in polyethylene lined hessian bags laminated with bitumen or in suitable containers as agreed to between the purchaser and the supplier.

**3.2 Marking** — The bags or the containers, as the case may be, shall be marked with the following:

- a) Name of the material;
- b) Manufacturer's name or trade-mark, if any;
- c) Net mass of the material; and
- d) Month and year of manufacture.

**3.2.1** The bags or the containers may also be marked with the ISI Certification Mark.

**NOTE** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

### 4. SAMPLING AND CRITERIA FOR CONFORMITY

**4.1 Sampling** — The sampling shall be done as prescribed in 15 of IS : 7086 ( Part I )-1973\*.

**4.2 Number of Tests and Criteria for Conforming** — Tests for all characteristics shall be conducted on composite sample. The lot shall be considered as conforming to the specification if the composite sample satisfies each of these requirements.

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\*Methods of sampling and test for rubber compounding ingredient, Part I.

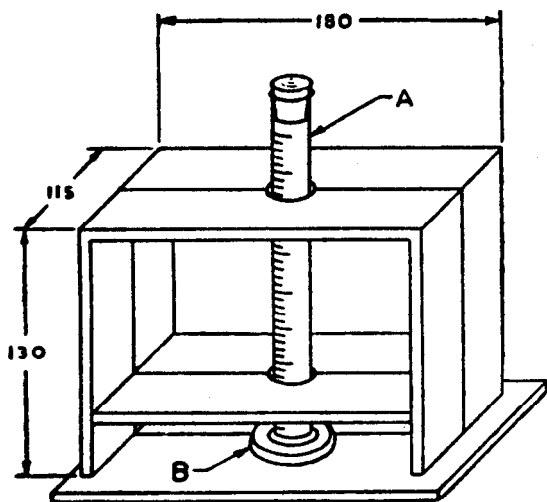
## APPENDIX A

[ Table 1, Item (i) ]

## DETERMINATION OF BULK DENSITY

## A-1. APPARATUS

**A-1.1** Assemble the apparatus as shown in Fig. 1. The base of the measuring cylinder *A* shall be ground flat and the empty measuring cylinder *A* together with the rubber bung shall weigh  $250 \pm 5$  g. It shall be accurately calibrated to 250 ml with an error, if any, of less than 1 ml. The distance between zero and 250-ml graduation on the measuring cylinder *A* shall be not less than 220 mm and not more than 240 mm. The distance between the flatground part of the base of the measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height shall be  $25 \pm 2$  mm.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF BULK DENSITY

**A-1.2 Rubber Base Pad** — The rubber base pad *B* shall have a hardness of 42 to 50 IRHD.

**A-1.3 Balance** — Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0.1 g.

## A-2. PROCEDURE

**A-2.1** Sieve about 40 g of the material through 250-micron IS Sieve on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 1. With the thumb and four fingers of one hand, gently grasp the upper part of the cylinder, and within one second lift it about 25 mm (taking a care not to jerk the cylinder by knocking it against the upper stop) and let it drop. Continue lifting and dropping until 50 complete drops have been given to the cylinder. During this operation give a gentle turn of about 10° in the clockwise direction to the cylinder after every two drops. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.

## A-3. CALCULATION

**A-3.1** Calculate the bulk density as follows:

$$\text{Bulk density, g/ml} = \frac{m}{V}$$

where

$m$  = mass in g of the material taken for the test, and

$V$  = volume in ml of the material after 50 taps.

# A P P E N D I X B

[ Table 1, Item (xi) ]

## DETERMINATION OF CALCIUM OXIDE

### B-1. REAGENTS

#### B-1.1 Concentrated Hydrochloric Acid

**B-1.2 Standard Ethylene Diamine Tetra Acetic Acid (EDTA) Solution** — N/50.

#### B-1.3 Murexide Indicator

**B-1.4 Ammonium Hydroxide-Ammonium Chloride Buffer Solution** — Mixture of 10 percent ammonium chloride and 30 percent ammonium hydroxide in the proportion of 1 : 5.

## B-2. PROCEDURE

**B-2.1** Digest about 0.5 g of the sample in 10 ml of concentrated hydrochloric acid in a 100-ml beaker, on a water-bath. Filter the solution after diluting it to 50 ml. Wash the residue collecting all the filtrate in a standard 250-ml flask. [ Preserve the residue for silica determination (see C-1.1) ]. Dilute the contents to 250 ml. Titrate 10 ml of this solution

against standard ethylene diamine tetra acetic acid ( EDTA ), solution using murexide as indicator and a mixture of ammonium chloride and ammonium hydroxide as buffer during titration.

### B-3. CALCULATION

$$\text{Calcium oxide, percent by mass} = \frac{14.02 VN}{M}$$

where

$V$  = volume in ml of standard EDTA solution used for the titration,

$N$  = normality of the EDTA solution used, and

$M$  = mass in g of the material taken for test.

## APPENDIX C

[ Table 1, Item (xii) ]

### DETERMINATION OF SILICA

#### C-1. PROCEDURE

**C-1.1** Transfer the residue on filter paper obtained in **B-2.1** to a weighed dry platinum crucible and ignite at 700°C in a muffle furnace for 2 hours and re-weigh to get silica content.

#### C-2. CALCULATION

$$\text{Silica ( as SiO}_2 \text{ ), percent by mass} = \frac{B - A}{M} \times 100$$

where

$B$  = mass in g of the crucible and residue after ignition,

$A$  = mass in g of the empty crucible, and

$M$  = mass in g of the material taken for test.

## APPENDIX D

[ Table 1, Item (xiii) ]

### DETERMINATION OF OIL ABSORPTION

#### D-1. REAGENTS

**D-1.1 Linseed Oil** — See IS : 75-1973\*.

\*Specification for linseed oil raw and refined ( *second revision* ).

**D-2. PROCEDURE**

**D-2.1** Place about 2 g of dry material accurately weighed, on a glazed porcelain, ground-glass or marble plate. Add linseed oil from a weighed dropping bottle, drop by drop, and regularly each drop being mixed well with the material using a knife. Incorporate the oil thoroughly in the course of 20 minutes, into the whole of the material with the knife, until a coherent mass is obtained. Weigh the dropping bottle again and determine by difference the mass of oil in grams. Where an approved sample is used for comparison, the oil absorption of the approved sample shall be determined by the same person and at the same time.

**D-3. CALCULATION**

**D-3.1** Oil absorption, g per 100 g =  $\frac{100 M}{M_1}$

where

$M$  = mass in g of linseed oil absorbed, and

$M_1$  = mass in g of the dry material taken for the test.

**A P P E N D I X E**

[ *Table 1, Item (xiv)* ]

**DETERMINATION OF SEDIMENTATION VOLUME****E-1. PROCEDURE**

**E-1.1** Transfer 2 g of the material in a stoppered graduated measuring cylinder. Make up the volume to 50 ml with toluene, shake thoroughly for 5 minutes and allow to stand for 1 hour. Measure the volume of the sediment.

**A P P E N D I X F**

[ *Table 1, Item (xv)* ]

**RETENTION IN SUSPENSION****F-1. PROCEDURE**

**F-1.1** Transfer 20 g of the material to a measuring cylinder, add sufficient water to make volume up to 100 ml and shake vigorously for 2 minutes. Allow to stand. Draw sample after 5 minutes and 20 minutes by means of 25 ml pipette from a point 10 cm below the surface of the liquid. Determine the mass per millilitre of the sample of suspension removed by means of a pycnometer.

( Continued from page 2 )

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